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PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Improvements in or relating to means for Imparting Durable Pleats, Creases or other Predetermined Deformations in Slivers Yarns or Fabrics consisting of or containing Wool or other Keratinous Fibres

We, WOOL INDUSTRIES RESEARCH ASSOCIATION, of Torridon, Headingley, Leeds 6, in the County of York, a Company limited by guarantee under the Companies Acts of 1908—17 and 1948, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention refers to the treatment of wool or other animal hairs, in the form of slivers or yarn, or of woven, felted or knitted fabrics, consisting of or containing wool or other keratinous fibres either in the piece or in the form of made-up garments (hereinafter referred to as "wool material"), to enable the durable creases, pleats or other predetermined deformations that will be maintained during wear and laundering or dry-cleaning treatments to be set in the wool material.

It is known that fabrics made from certain synthetic fibres may be deformed and then set in the deformed state by appropriate heat treatment and that this treatment confers useful properties on garments made from the fabric or on garments so treated. It is also known that deformation in wool materials cannot be set permanently by simple heat treatment and that a temporary set may be imparted by treatment with steam, e.g. in a steam pressing machine, or by hot water, e.g. in the crabbing or blowing processes. The temporary set thus imparted will, in general, be stable under conditions of use less severe than those used in imparting the set, e.g. material set in hot or boiling water will be stable in water at a lower temperature than that used in setting, whilst steam pressed fabric will tend to lose the set under wet conditions even at low temperatures. Other factors that tend to break down any set imparted by these means will include excessive relaxation of strain or felting together of the fabric caused by, e.g. alternate compression and relaxation as in a washing or a rubbing process. It has been shown that treatment of a curled yarn in boiling water for a suitable time, e.g. 4—6 hours, renders the curl stable to treatment in hot solutions, e.g. boiling dyebaths.

It is also known that treatment of keratinous fibres by hot water or steam as described above causes breakdown of the disulphide groups and allows a molecular rearrangement which is stabilised by a setting process that arises by the formation either of new cross-links of a co-valent type or of hydrogen bonds or similar electro-valent cross-linking.

Further it is known that the breakdown of the disulphide bonds can be more readily achieved by treatment with chemical reagents, especially those that cause reduction, such as organic and inorganic sulphites and hydrogen sulphite, and hydrazine. In order to allow or facilitate the subsequent setting processes, it is considered necessary to remove or "neutralise" excess of the disulphide-splitting reagents. Thus, the use of an oxidising agent, for example, hydrogen peroxide, has been described. Also described is the use of suitable reducing reagents, such as ammonium thioglycollate, which are volatile, or readily decomposed by heat to give volatile products.

It is also known that setting, following the rupture of disulphide bonds by a suitable reducing agent, can be achieved by treatment with a bifunctional cross-linking reagent such as ethylene dibromide. With the exception of this latter process, which is both costly and attended by disadvantages in its operation, the actual setting process results from the steaming and drying treatment that is applied as part

of the process described above. It can therefore be stated that with practically all the setting processes so far described the actual mechanism of the setting of any structural deformation is a matter for speculation and, according to current theories, resolves itself between formation of either new cross-linking that arises fortuitously by virtue of the random proximity of reactive groups or by hydrogen bonding of the rearranged long-chain molecules.

It follows from the above that any process for the purpose of rendering permanent or durable deformations in wool containing materials as described above would be facilitated and show enhanced efficiency by the application of (a) reagents that bring about the breakdown of hydrogen bonding and (b) reagents that are capable of forming a complex with a keratin and thereby producing a suitable form of intramolecular linking. These two considerations form the basis of the present invention.

According to the invention the process for the treatment of wool as hereinbefore defined in the form of slivers or yarn, or of woven, felted or knitted fabrics either in the piece or in the form of made up garments for the purpose of enabling durable creases, pleats or other predetermined deformations to be set into wool material, consists in treating the wool material with, a mixed solution containing (1) a reducing compound, (2) a hydrogen-bond breakdown reagent i.e. urea, guanidine, a phenol or formic acid *or* an anionic detergent e.g. a sodium, ammonium, magnesium, or calcium salt of an alkyl sulphuric acid or of an alkyl sulphonic acid or of an alkyl-aryl-sulphonic acid in which alkyl denotes a straight or branched hydrocarbon chain, preferably dodecyl, and aryl denotes a cyclic hydrocarbon ring, preferably benzene and (3) a pre-condensate of a resin together with an accelerator that facilitates the condensation of the resin pre-condensate, the final setting part of the process being accomplished by the application of moist heat with or without mechanical pressure.

We have also found that the incorporation into the treating solution of a pre-condensate of a resin, such as melamine-formaldehyde resin pre-condensate an accelerator that facilitates the condensation of the resin pre-condensate enhances the behaviour of the wool material, which has been submitted to conditions that bring about polymerization of the pre-condensate, during frequent launderings by giving to the wool material the property of retaining a clean surface together with freedom from cockles and waves.

In using the process of this invention, described above, the precise methods of application will depend on (a) the type of wool-containing material, e.g. whether sliver, yarn or woven, felted or knitted fabrics, and (b) the stage in the processing at which the methods will be applied, e.g. before or during making up of garments. These methods are exemplified below.

In the production of curled slivers or yarns, the mixed chemicals consisting of reducing compound, hydrogen-bond breaker or detergent, and the resin pre-condensate (containing an accelerator that facilitates condensation), are added to the rinsing water in the last bowl of the washing train, in such quantity that will leave a suitable concentration on the wool-containing material as it leaves the squeezing head attached to the washing machine. If the wool-containing material is to be curled or otherwise deformed immediately, the squeezing action must be controlled, or the condition of drying regulated, to leave 30%—60% (weight for weight) of the solution on the wool-containing material. The damp material is then curled or otherwise deformed, packaged or constrained so that the desired deformation is maintained and the whole is heated under suitable conditions. The best results are obtained by the use of a steam autoclave at 120°—125°C for a period of 5—30 minutes: the use of higher temperatures, which might be applicable to certain keratinous material, is not precluded from our process. If the material is to be curled or otherwise deformed at a later stage, it is dried at a low temperature, e.g. not exceeding 50°C; before deformation, in this case, the material is uniformly wetted by, e.g. spraying, with 30%—60% of moisture (weight for weight), deformed and set as described above.

In the production of durable creases and pleats, impregnation by the mixed reagents from a solution by means of e.g. a pad mangle, constitutes the final stage of any sequence of finishing operations; drying is carried out at a low temperature e.g. not exceeding 50°C. The sensitised fabric is then made into garments and the creases inserted during manufacture are set by moistening the garment with 30% to 60% (weight for weight) by e.g. spraying, and pressing by any conventional means, e.g. hand-iron or Hoffman press. By "sensitised" fabric we mean fabric that has been treated by the mixed reagents and then dried at a low temperature as described above. It is preferred to complete the treatment by heating the garments at a temperature of 110°—120°C for 5—10 minutes. It is also possible to manufacture garments from untreated fabric and, after creases have been inserted, spray a solution of the mixed reagents over the part of the garment containing the crease, or preferably, over the whole garment, so that the treated fabric contains the desired amounts of setting reagents and water as indicated above. The setting is then accomplished by the methods described above.

Similar methods are used for the insertion of pleats in skirt panels, but the preferred

method consists of moistening the "sensitised" fabric by spraying the panels with 30%—60% of water (weight for weight) before insertion between the male and female pleating formers, which are then rolled into bundles and held in position by appropriate means. For setting the pleats, the preferred method consists of the treatment of the bundled panels in an autoclave at a temperature of not less than 100°C for 5—30 minutes; a preferred technique includes the creation of a vacuum of not less than 25 inches of mercury before admission of steam.

In the setting of felted masses of fibres, e.g. in the production of felt caps that will not shrink during subsequent wetting, the preformed cap-bodies are impregnated by soaking in a solution of the mixed reagents and hydroextracting until the felt material contains the desired amounts of setting reagents and water. After pulling the bodies into the desired shape on a former the setting reactions are completed by the conventional heat-drying methods used in the Trade, e.g. on gas-heated metal formers.

The quantities of reagents that have been found to be necessary to ensure good stabilisation of deformations introduced into wool materials are as follows:

Reducing Compounds ½%—4% weight for weight on the wool material.

Hydrogen-bond-breaking Compounds ½%—10% weight for weight on the wool material.

Anionic Detergent 0.1%—2% (as alkyl or aryl sulphonate part of the compound) weight for weight on the wool material.

Resin 1%—5% (weight based on solid resin).

The following substances have been shown to give satisfactory results under the conditions indicated.

Reducing Compounds Alkali metal sulphites, bisulphites and metabisulphites: alkali metal hypophosphites: thioglycollic acid and its ammonium and sodium salts: sodium hydrosulphite or one of its derivatives or formamidine sulphinic acid (thiourea dioxide) and its soluble salts.

Hydrogen - bond - breaking Compounds Urea: guanidine: phenols: formic acid.

Anionic Detergent Sodium, ammonium, magnesium and calcium salts of alkylsulphuric or alkylsulphonic acid and alkylarylsulphonic acid, in which alkyl denotes a straight or branched hydrocarbon chain, preferably dodecyl, and aryl denotes a cyclic hydrocarbon ring, preferably benzene.

The process may also include the use of other anionic detergents and of normal soaps such as the alkali metal salts of alkylcarboxylic acids (where alkyl has the significance stated above.)

Resins Many condensation polymer resins are applicable to the processes described. The desiderata for their use include the resin being available in an appropriate dispersed and precondensed state and being readily polymerisable with or without the use of accelerators such as diammonium phosphate, magnesium chloride and zinc nitrate, at a temperature suitable to the wool material concerned.

Examples

In order to obtain an estimate of the durability of the pleat or crease during wear and/or laundering and/or dry cleaning, apart from visual observation, two methods of test are used, in combination with two methods of estimating the changes ensuing. These are as follows.

(a) *Testing and Durability*

Specimens of the pleated or creased fabric are:

either (i) Soaked in a solution of a non-ionic detergent (Lissapol N (R.T.M.) 0.1%) for about 17 hours the solution being initially at a temperature of 50°C (120°F) and being allowed to cool. After soaking, the specimens are rinsed in water, allowed to dry, without restraint at a temperature not exceeding 50°C (120°F) and then conditioned for at least 24 hours in the standard atmosphere (B.S.1051).

or (ii) Given 6 washes (each of 30 minutes duration and with occasional agitation) in a similar solution to that used in (i) above, the temperature being maintained at 50°C (120°F). After each wash, the specimens are rinsed and dried without restraint at a temperature not exceeding 50°C (120°F): after the 6th wash, the specimens are rinsed, dried and conditioned as in (i).

(b) *Estimation of the changes taking place in the tests.*

(i) *Pleated fabrics.* A strip of fabric 30cm × 10cm. is formed into pleats across the narrower dimension. The pleats are set by means of one of the methods described elsewhere in this specification and, after conditioning in the standard atmosphere (B.S.1051), the effective length of the strip is measured while it is hanging freely from one end, the pleats being in a horizontal position and allowed to open out naturally. The measurement is repeated after the tests (a) (i) and (ii) and the durability in each test calculated as follows:

Let the original length be Lo
" " final " " Lw
then Durability of pleating (%) is given by

$$\frac{30 - Lw}{30 - Lo} \times 100$$

(ii) *Pleated or creased fabrics.* A method similar to that described by Wilkinson and Ireland (*Text.Res. J.*, 1958, *28*, 674—679) is used. A sample of the pleated or creased fabric is cut 2 cm. × 1 cm. with the pleat or crease centrally across the shorter dimension and is then straddled across the horizontal edge of a razor-blade situated in front of a source of light. The V-shaped image of the pleat or crease is thrown on to a screen at a magnification of 9.5. The width of the crease (x) is measured on the image at a fixed distance from the apex on a vertical line through the apex. A further sample of the material is given the tests (a) (i) and (ii) and the measurement again made in the way described. An estimate of the crease sharpness (A) is given by the empirical relationship $\log A = 2 - Kx$ where K is an arbitrary constant. The crease-retention % (durability) is given by

$$\frac{\text{crease sharpness final state}}{\text{crease sharpness initially}} \times 100$$

The following examples illustrate, but do not limit, the invention.

Note: In the Examples all parts are by weight.

1. Worsted fabric (2/2 Twill: 2/48s yarn made from 64s wool: 72 ends and 70 picks per inch: 8.35 oz. per sq. yd. finished) 100 parts was impregnated by spraying with solutions of sodium bisulphite *or* sodium bisulphite and urea *or* sodium bisulphite, urea and resin (a commercial melamine-formaldehyde pre-condensate), with diammonium phosphate, 4% by weight on the resin, as accelerators so as to leave 50 parts of the solution on the fabric, the amounts of the reagents being given in Table I. The moist fabrics were then formed into pleats by wrapping alternately round thin metal strips held in a former, after which the metal strips were removed and the pleated fabric held in position by rubber bands. Alternatively, the pleats were formed by the use of pleating paper formers. The pleats were set *either* by treatment in an autoclave for 5 minutes at 10 lb steam pressure giving a temperature of 116°C (240°F), *or* in a Hoffman press using 50lb steam pressure allowing 20 seconds steaming time with the press closed, 20 seconds "baking" with the steam turned off and the press closed and 20 seconds vacuum with the press open to cool the fabric. The durability of the pleats was tested by methods (a) (i) and (ii) and (b) (i) described above. The results are given in Table I.

TABLE I

Sodium Bisulphite Parts	Urea Parts	Resin Parts	Setting Method	Pleat Retention (%)	
				Test (a) (i)	Test (a) (ii)
0	0	1	Autoclave	74	76
1	0	0	„	76	40
0	6	0	„	92	30
1	6	0	„	105	94
0	12	0	„	98	57
1	12	0	„	105	92
1	6	0	Hoffman	104	109
1	6	1	„	138	104
2	0	0	Autoclave	84	61
2	6	0	„	102	99
2	12	0	„	130	105
2	12	0	Hoffman	96	113
2	12	1	„	140	110

2. Lengths of worsted fabric of different structures and weights each 100 parts were impregnated by a padding technique with sodium bisulphite (2 parts), sodium dodecylbenzene sulphonate, straight-chain (1 part) and melamine-formaldehyde resin pre-condensate (2 parts) including diammonium phosphate (0.08 parts) as an accelerator for the resin. The fabrics were dried at a low temperature (20°C) and after 3 weeks were made into trousers, the creases being set by the use of a wet cloth and a Hoffman press. After hanging up to dry, the trousers were heat-treated in a Hoffman press for 20 seconds (steam: 70lb/in²). After 6 months wear the creases were still sharp.

3. Lengths of worsted fabric of different structures and weights, each 100 parts, were treated as those in Example 2 and after 4 weeks were made into skirt-panels that were accordian-pleated and set by heat-treatment in an autoclave at 30lb pressure for 10 minutes. After making into skirts, the garments were worn for 6 months, during which time they were dry-cleaned once, washed in a vertical type domestic electric washing machine and dried by hanging without restraint at a low temperature (20°C). The pleats were reasonably sharp and were restored to the original condition by mild steam treatment in a free state.

4. Further lengths of worsted fabric (plain weave: 2/38s yarn from 64s wool: 70 ends and picks per inch) were treated as those in Example 2 and after 2 months storage were made into fan-pleated skirt panels and set by the method used in Example 3. The pleated panels were dry-cleaned 5 times being finally hung freely in steam and the durability of the pleats estimated by hanging each panel freely from one horizontally positioned edge so that the panel assumed a fan-like shape; the angle (θ) was then measured between the two sides of the panel. This measurement was made after setting and after each of the dry-cleaning treatments. The results are given in Table VI.

TABLE VI

Treatment	Angle of pleated panel θ°
After setting	70
1st dry clean	70
2nd dry clean	73
3rd dry clean	80
4th dry clean	80
5th dry clean	81
Hung in steam	72

WHAT WE CLAIM IS:—

1. A process for the treatment of wool as hereinbefore defined in the form of slivers or yarn, or of woven, felted or knitted fabrics either in the piece or in the form of made up garments for the purpose of enabling durable creases, pleats or other predetermined deformations to be set into wool material, wherein the wool material is treated with a mixed solution containing (1) a reducing compound, (2) a hydrogen-bond breakdown reagent i.e. urea, guanidine, a phenol or formic acid *or* an anionic detergent e.g. a sodium, ammonium, magnesium, or calcium salt of an alkyl sulphuric acid or of an alkyl sulphonic acid or of an alkyl-arylsulphonic acid in which alkyl denotes a straight or branched hydrocarbon chain preferably dodecyl, and aryl denotes a cyclic hydrocarbon ring, preferably benzene, and (3) a pre-condensate of a resin together with an accelerator that facilitates the condensation of the resin pre-condensate, the final setting part of the process being accomplished by the application of moist heat with or without mechanical pressure.

2. A process as claimed in Claim 1, in which the reducing compound is one or more of the following: an alkali metal sulphite, or metabisulphite: an alkali metal hypophosphite: thioglycollic acid or its ammonium or sodium salt: sodium hydrosulphite or one of its derivatives: formamidine sulphinic acid (thiourea dioxide) and its soluble salts.

3. A process as claimed in Claim 1 where the resin is a melamine-formaldehyde in a precondensed state together with the appropriate accelerator, e.g. diammonium phosphate, magnesium chloride and zinc nitrate.

4. A process as claimed in any of the Claims 1 to 3, in which the wool material is deformed and set by the application of steam and mechanical pressure in a Hoffman press or by a hand-iron.

5. A process as claimed in any of the Claims 1 to 3 in which the wool material is deformed and set by the application of steam at a temperature exceeding 120°C.

6. A process as claimed in any of the Claims 1 to 3 in which the wool material is deformed and set by the application of steam by conventional heat drying methods, e.g. a heated metal former.

7. A process as claimed in any of the preceding Claims substantially as described with reference to any of the Examples given.

8. Wool material as hereinbefore defined which has been treated by the process claimed in any one of the preceding Claims.

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